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Executive Summary Report

The Use of Atomic Force Microscopy
To Evaluate Warm Mix Asphalt

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Problem Statement

Warm Mix Asphalt (WMA) has received considerable attention in past few years due to its benefits in reducing energy consumption and pollutant emissions during production and placement of asphalt mixtures, as well as a compaction aid during the construction process. However, many concerns and questions are still unanswered regarding the performance and durability of WMA. One key issue is the moisture susceptibility of WMA. Although the results of standard laboratory tests indicated that WMA may be more susceptible to moisture damage than Hot Mix Asphalt (HMA), data obtained from the field does not support those results. Some data also suggests that the resistance of WMA to moisture damage improves with time and may ultimately be equivalent to that of HMA. In addition, the healing characteristics of WMA have not yet been studied nor evaluated in a methodical, scientific manner. Therefore, research is needed to determine if the degree of healing in WMA is sufficient to increase its resistance to damage, and hence enhance their long term durability.

The fundamental understanding and evaluation of the moisture damage and healing characteristics of WMA requires careful consideration of the micro-mechanisms that influence the adhesive bonds between the asphalt binder and the aggregate, and the cohesive bonds within the asphalt binder. However, all standard laboratory tests that have been used to evaluate the WMA examine their integral, macro-scale behavior only. Therefore, those tests are limited in their ability to validate the moisture damage and healing mechanisms in an asphalt system, as they cannot examine and determine factors contributing to its response at the micro-scale.

This research aims at investigating a paradigm shift in the characterization of WMA such that the recent progress in nano-mechanics and material science can be utilized to study the moisture damage resistance and healing characteristics of WMA, and compare them to those of conventional HMA. The novelty of the pursued approach is to examine the nano-scale behavior for WMA using the Atomic Force Microscopy (AFM) and to identify nano-scale parameters that can be used to interpret the macro-scale field performance. The practical outcome of this project is an improved nano-to-macro understanding of WMA that can facilitate the successful implementation of WMA in Ohio, and lead to the development of sustainable pavement structures.

Study Objectives

The main objective of this research project is to study the micro-scale behavior of WMA using the AFM. The specific objectives of this study include:

- Develop sample preparation procedures and testing protocols for nano-scale AFM testing on asphalt materials.
- Identify the AFM testing parameters that can be utilized in the evaluation of moisture susceptibility and healing characteristics of asphalt materials.
- Quantify the nano-scale level adhesive and cohesive forces in a WMA system under dry and wet conditions using AFM and compare it to that in a control HMA.
- Evaluate the influence of WMA technologies on the healing characteristics of asphalt binders.

Description of Work

To achieve the objectives of this study, AFM tapping mode imaging, force spectroscopy, and healing experiments were conducted on two types of asphalt binders (PG 64-22 and PG70-22M) produced using various WMA technologies as well as a conventional HMA. The considered WMA technologies included: Advera, Evotherm M1, Sasobit, and foamed WMA. Dynamic Shear Rheometer (DSR) tests were also conducted on the evaluated binders, and the AASHTO T283 test was performed on mixtures prepared using those binders.

Two methods were evaluated to prepare asphalt samples for AFM testing. The first method was found to be the optimum one to form uniform and consistent surfaces required for all AFM characterization techniques. AFM imaging was performed on the prepared asphalt samples using tapping mode to characterize their nano/micro-structure. The AFM tapping mode imaging technique was used to minimize sample deformation and avoid the surface and/or tip damage found in contact mode AFM. In this technique, the AFM cantilever/tip system is oscillated at its resonant frequency and the piezo-driver is adjusted using feedback control to maintain a constant tip-to-sample distance (set-point). A relatively high set-point of 88% and free amplitude of 240 nm were used in this study to detect the discrepancies in the viscoelastic properties within the asphalt binders and map out domains with different viscoelastic properties.

AFM force spectroscopy experiments were also employed to measure the nano-scale adhesive and cohesive forces in the asphalt systems before and after moisture conditioning to examine the moisture susceptibility of WMA and compare it with that of control HMA material. Force spectroscopy experiments were conducted with silicon nitride tips to examine the adhesive forces between the asphalt and gravel aggregate. Furthermore, the interaction forces between asphalt molecules (i.e. cohesive forces) were examined by using tips that are chemically functionalized by two of the main chemical groups found in asphalt binders, namely hydroxyl (-OH) and carboxyl (-COOH) groups.

An AFM-based approach was also employed in this study to examine the healing characteristics of HMA and WMA binder. The employed approach evaluates the two mechanisms of healing: wetting and intrinsic healing. The wetting mechanism is studied by probing (indenting) the asphalt sample using the AFM tip at a fixed location and indentation depth, to create a nano-crack in the sample. AFM images are then continually taken to record the asphalt crack recovery with time. The images are post-processed and analyzed to measure the closure of the initiated crack with time, which is used to evaluate the wetting rate for the tested asphalt material. In addition, the intrinsic healing is evaluated by determining the energy required to overcome the cohesion bonds within the asphalt material, referred to as the cohesive bonding energy, by utilizing the results of the force spectroscopy experiments performed using tips functionalized with –OH and –COOH chemical groups.

Research Findings & Conclusions

Based on the nano and macro-scale test results and the subsequent statistical analyses findings, the following conclusions were made:

- The AFM was found to be a viable device to examine the moisture damage mechanisms by conducting force spectroscopy experiments using chemically functionalized tips that resembles the aggregates particles and asphalt molecules and measuring the adhesive and cohesive forces.
- The AFM is an effective tool to examine the two main healing mechanisms in asphalt materials, namely, wetting and intrinsic healing.
- Based on the AFM imaging results, the Sasobit additive resulted in increasing the relative stiffness of dispersed domains containing the 'bee-like' structure in comparison with the flat asphalt matrix for both types of asphalt binders. The stiffer domains resulted in enhancing the stiffness properties of the Sasobit modified asphalt binder as compared to the control asphalt binder, which explains the higher shear modulus values obtained in the DSR test for this binder.
- The AFM phase image showed that the inclusion of Evotherm did not have a significant effect on the domains viscoelastic properties of the PG 70-22M and PG 64-22 binders. This is consistent with DSR test results.
- According to the AFM phase image, the foaming of the polymer modified asphalt binders through adding Advera additive or by water injection resulted in reducing the relative stiffness between the dispersed domains and the flat asphalt matrix.
- Based on the results of the AFM force spectroscopy experiments, the indirect tensile strength (ITS) measured in the AASHTO T283 test was found to depend more on the adhesive bonds between the aggregate and binder as compared to the cohesive bonds within the binder itself.
- For the unconditioned samples, all WMA technologies had resulted in increasing the nano-scale adhesive forces for both types of asphalt binders. Advera and foamed WMA had the highest improvement to these forces, while the Sasobit had the least. This may explain the lower ITS values obtained due to the addition of the Sasobit.
- For the unconditioned 70-22M samples, the inclusion of the Evotherm and Advera WMA additives resulted in significantly increasing the –OH interaction forces, but the Sasobit additive significantly reduced these forces.
- For the unconditioned samples, only the Sasobit has resulted in significant reduction in the –COOH cohesive forces for both types of binders. However, the other WMA technologies did not have any significant effect on these forces.
- While the conditioned Advera, Evotherm, and foamed WMA had similar adhesive force values to that of the conditioned control PG 70-22M binder, the Sasobit possessed significantly lower value.
- The conditioned Evotherm and foamed WMA had statistically similar –COOH and –OH interaction forces to that of the conditioned control PG 70-22M binder. On the contrary, the conditioned Advera and Sasobit had statistically lower cohesive force values than the conditioned control binder.

- The adhesive forces were significantly decreased due to conditioning of the HMA and WMA 64-22 binders. This behavior was different than that observed for the polymer modified 70-22M binders, which indicates that the 64-22 binders are more susceptible to moisture damage.
- Upon conditioning of the PG 64-22 binder, the control and Evotherm WMA binders exhibited the least reduction in the adhesive forces. Furthermore, the Advera binder had the highest decrease after moisture conditioning. This may explain the lower TSR values that Advera 64-22 mixture exhibited.
- The Sasobit and Advera led to a reduction in the cohesive forces within both types of asphalt binders after moisture conditioning, indicating that it might adversely affect cohesive bonds within the asphalt binder.
- The Evotherm, Advera, and foamed WMA technologies had resulted in improving the rate of micro-crack closure rate in the two types of binders considered in this study. On the contrary, Sasobit had an adverse effect on this rate.
- The Sasobit resulted in significant decrease in the cohesive bonding energy; indicating that it might adversely affect the intrinsic healing of the considered asphalt binders.
- Advera and foamed WMA technologies do not have a significant effect on the intrinsic healing of the considered asphalt binders.
- The Evotherm improved the intrinsic healing of the two types of asphalt binders considered in this study.

Study Limitations

- The use of only two types of asphalt binders and one type of aggregates.
- The use of fully dried aggregates in preparing the asphalt mixtures.
- Only one type of macro-scale test was used in this study to evaluate moisture susceptibility of asphalt mixtures.
- The foaming parameters were not varied and thus their influence on moisture susceptibility was not investigated.

Recommendations for Further Study

- It is recommended that future work expands the current study to include other types of aggregates and asphalt binders used in Ohio and to study the effects of the foaming parameters on the moisture susceptibility of WMA binders and mixtures.
- Future studies are needed to evaluate the use of other macro-scale tests that can examine the adhesive as well as cohesive failures within an asphalt mixture.
- Future research is recommended to explore the use of various AFM techniques to solve other problems related to asphalt pavements.

Recommendations for Implementation

- It is recommended to use an additional test to supplement the AASHTO T283 test for examining moisture-induced damage problems in asphalt mixtures associated with cohesive bonds failures.
- The foamed WMA technology was not found to have any significant effect on the moisture susceptibility of the considered asphalt binders. The foamed WMA technology may also improve the micro-crack closure rate of asphalt binders, but it does not affect their intrinsic healing properties.